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DEVELOPMENT OF THE FIELD **FUEL QUALITY MONITOR**

INTERIM REPORT AFLRL No. 184

By

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FOREWORD

The work presented herein was conducted at the U.S. Army Fuels and Lubricants Research Laboratory (USAFLRL), Southwest Research Institute, San Antonio, Texas, under Contracts DAAK70-78-C-0001, DAAK70-80-C-0001, DAAK70-82-C-0001, and DAAK70-85-C-0007. The work was funded by the Belvoir Research and Development Center, Ft. Belvoir, VA. Contracting Officer's representative was Mr. F.W. Schaekel, and the technical monitor was Mr. M.E. LePera, Fuels and Lubricants Division, Materials, Fuels, and Lubricants Laboratory (STRBE-VF).

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I. INTRODUCTION

Long-term storage stability and cleanliness have been requirements imposed on military fuels designed for tactical/combat environments. These requirements have been necessary to ensure that fuels delivered or deployed in strategic locations will be satisfactory for use during combat operations regardless of the time interval between refinery production and ultimate use. This need becomes very critical in relation to prepositioning of fuel stocks in which good fuel quality must be maintained. Without guaranteed maintenance of fuel cleanliness and stability during combat operations, combat fuels could lead to equipment vulnerability, engine malfunctions, increased maintenance, decreased mobility, as well as numerous other problems.

Table 1 lists those major thrust areas which have been identified in the Army's distillate fuel stability program. This table has provided the basis for a discussion of Army needs for diesel fuel stability and cleanliness in a report distributed in 1980. (1)*

The current report details the development of a field fuel quality monitor for the stability and cleanliness of distillate fuel to address the first thrust area, "Field Test Technique Development," given in Table 1.

TABLE I. MAJOR THRUST AREAS IN ARMY DISTILLATE FUEL STABILITY PROGRAM

	Thrust Arcas	Purpose
•	Field Test Technique Dévelopment	Test quality and predict future quality
•	Survey Quality of Petroleum Fuels	Correlate laboratory methods, specifications, field manuals
•	Conduct Field Problem Surveillance	Identify fuel-related problems and adequacy of fuel procurement and handling practices
	Evaluate Additives for Depot Use and Prepositioning	Provide fuel additive package for dormant storage of fully fueled vehicles
•	Conduct Basic Research	Define factors leading to deleterious products in both short- and long-term storage

^{*}Underscored numbers in parentheses refer to the list of references at the end of this report.

IL BACKGROUND

Quick, simple, and most importantly, reliable methods of detecting excessive fuel contamination while in the field have long been sought. Although field labora ories offer a solution to this problem, they are usually large (requiring a vehicle to transport them) and expensive to operate and maintain. A more practical solution is a small, self-contained portable kit that would furnish a go/no-go answer with regard to any given supply of fuel. Kits of this type are available commercially. One such device is the Fuel Contamination Detector (NSN: 2H6630-706-2302).(2) This kit, which has a self-contained vacuum pump to filter the fuel, uses a photovoltaic cell to measure the light that passes through the filter to determine the amount of contamination on the filter. The unit was developed for turbine fuel and has not been fully evaluated for diesel fuel; however, the Navy has evaluated it for use with marine diesel fuel. The Navy concluded that the device could be used to give a no/no-go answer.(3) This kit, however, requires an AC power source, a major disadvantage for a field-operated device.

The Army developed and type-classified standard "A", an Aviation Fuel Contamination Test Kit in May 1984. Designed primarily for field use, the kit can conduct three basic tests: determination of the free (undissolved) water content by Aqua-Glo method, particulate contamination by color standard, and fuel identification by gravity using API hydrometers. Also available in the kit are matched weight filters. These filters are available for taking particulate contamination samples in the field for later gravimetric determination on an analytical balance in a Base Laboratory, Mobile Laboratory, or other suitable laboratory facility.

Another test used for determining fuel cleanliness is the Filterability Ratio Test (see Appendix A). This procedure has been used with considerable success in the laboratory in predicting potential fuel deterioration/cleanliness problems. In this test, the time required to filter a given amount of contaminated fuel is compared to the filtering time for an equal portion of the same fuel that has been prefiltered. A value of 1.0 for the test indicates a perfectly clean fuel. However, this test must have a constant vacuum source, which is sometimes difficult to obtain in a field situation.

In addition to testing for cleanliness, it is desirable to rate a fuel's approximate storage stability. The U.S. Army Fuels and Lubricants Research Laboratory (AFLRL) has an ongoing program devoted to the development and evaluation of an in-the-field fuel cleanliness/stability testing device.

A laboratory study was initiated in late 1976 to define the relative stability characteristics of a base set of fourteen test fuels. This study was used as input to the definition of a field test capability for measuring quality and predicting stability. Table 2 summarizes particulates data for seven test fuels using five accelerated test procedures described in Appendix A. Note that the particulates in the 150°C, 80°C, and 43°C methods were determined using ASTM Method D 2276, modified to incorporate a 1.2-micrometer pore size membrane filter.

TABLE 2. DISTILLATE FUEL STABILITY DATA BASE—PARTCULATES BY VARIOUS ACCELERATED STORAGE TESTS

. Stability		Partleu	dates, mg/100 m Fuel Sam	il, D 2276 (m ole Number	iod., 1.2µm)	
Test	i	2	3	4	5	ő	7
•D 2274	0.2	0.3	0.3	0.7	1.1	1.8	5.3
●D 873	0.4	1.6	1.2	0.9	2.8	5.1	19.9
•150°C, 90 min	1.0	3.4	1.0	1.6	2.0	2.6	4.3
-80°C, 72 hr	0.8	1.2	0.3	0.4	0.4	1.7	1.4
168 hr	0.6	1.7	0.4	1.0	1.7	3.5	4.1
"43°C, lowerks	0.8	0.6	4.1	0.2	0.2	3.6	0.4
32 weeks	0.8	0.5	S. (1.1.) 75.	0.5	0.2	5.5	1.4

Based on data correlations of the fuels ranked according to particulates and sediment at 43°C (110°F) storage for 32 weeks, the best correlations were with the 30°C (175°F) test data. The D 2274 test data correlation was particularly poor. The test with the greatest potential for field use, based on its simplicity and short test time (90 minutes), was the 150°C (300°F) test tube method. Unfortunately, the 150°C test data correlated poorly for the ranked fuels. However, this method may offer some potential if a go/no-go value of 4.0 mg/100 mL is used. The data given in Table 3 for a number of field samples, reinforce the suitability of the 4.0 mg/100 mL value for go/no-go determination of fuel quality.

TABLE 3. COMPARISON OF ASTM METHOD D2274 AND 150°C TEST DATA FOR FIVE FUELS)

		Particulates, mg/100 ml							
Method	Fuel 1*	Fuel 2*	Fuel 3*	Fuel 4**	Fuel 5**				
D 2274	4.9	3.2	3.2	5.7	0.9				
150°C, 90 min	11.3	8.6	6,0	10.2	2.6				

^{*} Vehicle fuel cell samples

Federal specification VV-F-800C for diesel fuel limits the D 2274 value to 1.5 mg/100 mL total insolubles; hence, the values for fuels 1 through 4 in Table 3 are high both with respect to D 2274 and the test at 150°C for 90 minutes. The values in Table 4 were recently presented (5) as go/no-go values based on test results for

TABLE 4. FUEL STABILITY RATINGS OF GOOD, MARGINAL, AND BAD BASED ON LIMITING INSOLUBLES VALUES

	Limiting Values for Rating Fuels (mg/100 ml, slass fiber filters)							
Test Method	Good	Marsinal	Bad					
D 2274	<1.5	1.5-2.2	>2.2					
43.3°C Vented	ese sur la s La sur la sur							
4 wk	<2	2-3	>3					
5 wk	<2	2-3	>)					
16 wk	<2	2-3	>3					
24 wk	<2	2-3	>3					
150°C	•							
1.5 hr	<1		>6					
3.0 hr	<4	4-6	>6					
4.5 tir	<4	4-6	>6					
80°C								
3 days	<2	2-3	>3					
7 days	<2	2-3	>3					
14 days	<4	4-6	>6					
D 873	<4	4-6	>6					
93.3°C	<1.5	1.5-2.2	>2.2					

^{**} Bulk underground storage samples

six fuels in a program to develop accelerated stability test techniques for middle distillate fuels. For judging inherent fuel stability, total insolubles limiting values were selected (Table 4) for each of the six test methods. Fuels can then be rated as good, marginal, or bad if their insoluble values are less than the selected limiting value, less that 150 percent of the limiting value, or greater than 150 percent of the limiting value, respectively. Note that the limiting values for good, marginal, and bad for the 150°C test are <4, 4-6, and <6 mg/100 mL total insolubles, respectively.

III. LABORATORY PROTOTYPE FUEL QUALITY MONITOR

Basically an in-the-field fuel cleanliness/stability testing device (fuel quality tester) was envisioned to perform four functions:

- (1) sample the fuel from its storage container
- (2) rate the fuel for cleanliness,
- (3) age the fuel in some manner, and
- (4) rate the aged fuel for stability.

Methods of performing these functions were first evaluated in laboratory approaches prior to fabricating a field test unit.

A. Experimental

The laboratory prototype fuel quality tester was evaluated using nine fuels which had been in ambient, vented drum storage for 3 years. The 55-gallon drums containing the test fuels were each moved to a shaded area and stirred for 15 minutes with an air-driven stirrer. The drums were then allowed to stand for 2 days to allow the water to settle out. After this 2-day period, approximately 4 gallons of each fuel were sampled into a 5-gallon can. Each 5-gallon can was then used to simulate in-the-field fuel storage containers. Some of the unfiltered fuel was sent to the laboratory for standard tests, which included color, steam jet gum, water and sediment, total acid number, light absorbance, accelerated stability by ASTM Method D 2274, and particulates by ASTM Method D 2276 (modified). In addition, the filters from the D 2276 test were also rated using the prototype filter testing device. Table 5 is a compilation of this laboratory data. Examination of this data shows that the fuels used covered a wide range of stability, as measured by ASTM Method D 2274, but a more narrow range of cleanliness.

Figure 1 is a flow chart for the evaluation procedure. Note that there are two categories under which the evaluation procedures fall: "To Estimate Stability" and "To Measure Cleanliness". The device shown in Figure 2 was used to obtain a sample of the neat fuel and consisted of a plastic tubing long enough to sample at the desired depth in the fuel container, a filter holder, a receiving flask, and a small hand-operated pump used as a vacuum source. No filters were placed in the

TABLE 5. AMBIENT STORAGE OF DISTILLATE FUELS DATA AFTER 36 MONTHS STORAGE

Sample Code No.	6574	6624	6625	6626	6630	6638	6678	6716	6746
Particulates, D 2276, 1.2 μm , mg/500 ml	4.78	13.10	1.00	1.36	15.58	0.04	3.68	5.22	1.10
Accelerated Stability, D 2274, Total Insolubles, mg/100 ml	7.4	0.6	2.0	0.2	2.1	0.1	1.0	1.5	0.3
Steam Jet Com, D 381, mg/100 ml	11.4	32.2	4.0	4.2	40.3	0.2	7.9	13.2	2.2
Color (D 1500)	1.0	3.0	1.0	2.0	6.0	0.5	4.0	3.0	0.5
Total Acid No., D 664, mg KOH/gm sample	0.026	0.140	0.085	0.009	0.040	0.016	0.085	0.039	0.063
Light Absorbance at 650 nm 575 nm 540 nm 500 nm	0.021 0.048 0.073 0.128	0.159 0.259 0,346 0,520	0.001 0.010 0.026 0.059	0.015 0.060 0.107 0.191	0.292 0.645 1.132 2.070	0.001 0.001 0.001 0.001	0.075 0.185 0.372 0.831	0.041 0.116 0.193 0.327	0.001 0.001 0.001 0.007
Water & Sediment (D 1796)	A	В	None	None	C	None	None	A	None

A = trace sediment; no water.

Obtain a Sample of Neat Fuel Using the Field Filtering Device Without Filters

To Estimate Stability

- 1. Run color on the unfiltered fuel using the Oil Comparator
- 2. Age the unfiltered fuel at 150°C for 3 hours
- 3. Filter the aged fuel using the field filtering device
- 4. Rate the test filters using the Electro-Optic Photometer
- 5. Run color on the filtered fuel using the Oil Comparator

To Measure Cleanliness

- 1. Filter the unfiltered fuel using the field filtering device
- 2. Rate the test filters using the Electro-Optic Photometer
- 3. Run color on the filtered fuel using the Oil Comparator

FIGURE 1. FLOW CHART FOR EVALUATION OF FIELD FUEL QUALITY MONITOR

B = trace sediment; 0.30 vol water.

C = 0.1 vol% sediment; no water.

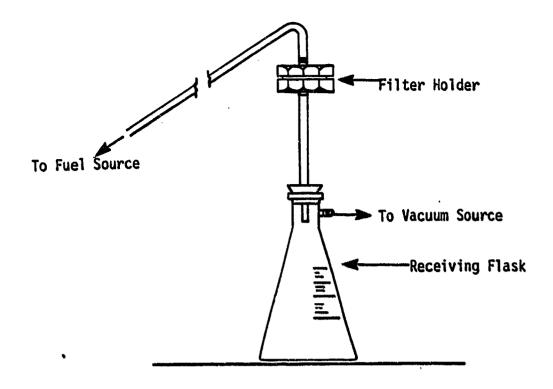


FIGURE 2. PROTOTYPE FIELD FILTERING DEVICE

filter holder while obtaining a sample of the neat fuel. To measure the cleanliness of the fuel, filters were installed in the field filtering device. The filters used during evaluation of the initial laboratory prototype device were 1.2-micrometer pore size cellulose acetate-cellulose nitrate membrane filters. The filter holder was manufactured by Schleicher and Schuell, Inc., Model No. FP050/9.

For this effort, 500 mL of the unfiltered fuel were filtered through two sandwiched 1.2-micrometer millipore filters (a sample filter and a control filter) using the prototype field filtering device (Figure 2). These filters were preweighed so they could be reweighed at a later time for gravimetric quantitation of particulates.

To rate the filters for cleanliness, an electro-optic photometer (EOP) was mocked up, since the feasibility had been qualitatively demonstrated previously. This instrument (Figure 3) projects a beam of light through the test filter and measures the amount of light that passes through the filter by means of a photosensitive cell. The lamp and detector of the EOP are shielded from external light during measurements.

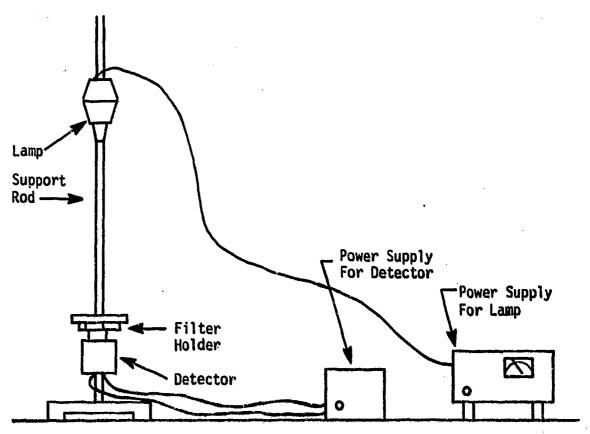


FIGURE 3. DIAGRAM OF LABORATORY PROTOTYPE ELECTRO-OPTIC PHOTOMETER APPARATUS

The amount of light blocked from passing through the filter is a function of the amount of particulate contamination present on the filter. To measure the color of the fuel, a commercial device manufactured by Hellige Incorporated called a Hellige Oil Comparator Outfit No. 607-OC was used. This device allows for the comparison of the color of the fuel to a set of standard colored discs. Detailed operating instructions of this equipment are found in Appendix B. To estimate the stability of the fuels, 55 mL of each fuel were prefiltered and placed into each of four test tubes to be heated at 150°C for 3 hours. After aging, the fuels in two of the tubes were filtered using the field filtering device (see Figure 2) with two preweighed 1.2-micrometer membrane filters. The fuels in the other two tubes were filtered by modified ASTM Method D 2276, also using 1.2-micrometer membrane filters. The field filtering device test filters and the D 2276 filters were then rated using the EOP. The filtered fuel from the field filtering device

was tested for color. The 1.2-micrometer filters from the D 2276 determination (both the 3-year storage samples and 150°C aged fuel) were rated by the EOP in a dry state. The field filtering device filters were rated while still wet with fuel.

B. Discussion

The hand pump used with the field filtering device was able to create enough suction to filter most of the fuels. However, if water was present in the fuel, filter plugging often occurred. In some cases, when using the field filtering device, the particulates tended to localize on a given area of the filter. The fuel was also occasionally permitted to flow either around or between the filters because the filter holder did not hold the filters securely in place and the vacuum was not constant. Each of these occurrences can result in erroneous readings when the filters are rated using the EOP. Table 6 lists data for the filtration of fuel through the field filtering device from both the filtration of the neat fuel and the filtration of the aged samples from the 150°C test. The table gives the weight of particulates left on each filter following filtration.

TABLE 6. DATA FOR FILTRATION OF FUEL THROUGH THE FIELD FILTERING DEVICE (1.2-micrometer pore size membrane filter)

						•		•	-
Sample Code No. Neat Fuel	6574	6624	6625	6626	6630	6638	6678	<u>6716</u>	6746
Weight of Particulates, mg/	lm 00i						٠,		
Sample filter	4.74	•	1.04	0.46	19.32	10.04	12.66	15.64	11.38
Control filter	1.46		1.00	0.08	12.66	10.02	12.00	8.14	10.08
Difference	3.28	•	0.04	0.38	6.66	0.02	0.66	7.50	1.30
150°C (3 hr Aging of Fuel)				i e					
Weight of Particulates, mg/	100 ml								
Sample filter	13.96	6.00	3.08	1.28	9.20	0.12	4.12	25.20	1.44
Control filter	0.12	0.24	0.08	0.28	0.60	0.12	0.00	0.28	-0.06
Difference	13.84	5.76	3.00	1.00	8,60	0.00	4.12	24.92	1.50

Sample would not filter due to high percentage of water present in fuel.

The EOP uses attenuation of a light beam by the test filter to estimate the amount of particulate contamination present on the filter. The amount of light that passes through the test filter is measured by a photosensitive cell. The percent light transmittance (%T) through each test filter was calculated using the formulas given in Table 7. The absorbance of each filter (sample and control) was calculated using transmittance values.

TABLE 7. TRANSMITTANCE/ABSORBANCE CALCULATIONS FOR TEST FILTERS

Transmittance, T, is defined as

 $T = I_T/I_0$

where

I_o = Initial Intensity
I_T = Transmitted Intensity

%T = T(100)

Absorbance, A. is defined as

 $A = \log 1/T = \log I_0/I_T$

Particulate Absorbance is equal to A of sample filter minus A of control filter

Particulate absorbance, the absorbance due to the particulate on the sample filter, is determined by subtracting the absorbance of the control filter from the absorbance of the sample filter.

The presence of particulates on the control filter (second or backup filter in each set) indicates that fuel was allowed to flow between the two filters. When this control filter weight is subtracted from the sample filter weight, the result is an erroneous weight for the total amount of particulates. Since the EOP reads the amount of particulates on the filter, the EOP readings would also be misleading if fuel were allowed to flow between the filters. One possible way to alleviate this problem is to use a filter holder similar to the type used in the ASTM Method D 2276. In D 2276, the two parts of the holder are clamped together, rather than

screwed; another solution might be to use a standard vacuum pump that has been converted to run on batteries rather than a hand pump. This vacuum pump would conceivably deliver a greater and more constant vacuum, thus reducing the possibility of the two filters separating and allowing fuel to flow between them.

For evaluation and calibration purposes, it was necessary to determine the relation of the EOP results to the actual weight of the particulates on the filter. EOP readings were recorded for each filter, and absorbance values were calculated. These absorbances were then converted to particulate absorbance values (Table 7) and compared to the weight of particulates as determined gravimetrically. Table 8 lists the various particulate absorbance values compared to the gravimetrically determined particulate weights.

TABLE 8. COMPARISON OF PARTICULATE ABSORBANCE VALUES TO ACTUAL PARTICULATE WEIGHTS

	Re	sults for Samples Fil	tered Using the Field						
	Neat Fuel, 1.2		150°C (3 hr aging), 1,2 μm						
Sample No. 6574	Particulate Absorbance 0.1	Particulate Weight, mg 3.28	Sample No. 6574	Particulate Absorbance 1.0	Particulate Weight, mg 13,84				
6624	•	•	6624	0.4	5.76				
6625	0.1	0.04	6625	0.1	3.00				
6626	0.1	0.38	6626	0.1	1.00				
6630	0.7	6.66	6630	1.0	8.60				
6638	. 0.1	0.02	6638	0.1	0.01				
6678	0.6	7.50	6678	0.1	4,12				
6716	0.1	1.30	6716	1.3	24.92				
6746	0.2	0.66	6746	0.1	1.50				

Results for Samples Filtered Using ASTM Method D 2276 (Modified) Neat Fuel, 1.2 um 150°C (3 hr aging), 1,2 um Particulate Particulate Particulate Particulate Sample No. Absorbance Weight, mg Samule No. Absorbance Weight, mg 6574 0.7 4.8 6574 0.5 7.4 6624 0.8 13.1 6624 1.1 5.8 6625 0.01 1.0 6625 0.2 3.4 6626 0.1 1.4 6626 0.1 1.2 6630 1.9 15.6 6630 1.6 11.1 6638 0.1 0.1 6638 0.1 0.1 6678 0.6 3.7 6678 0.5 5.0 6716 0.7 5.2 6716 1.7 25.8 6746 6746 0.1 1.2

^{*}Filter plugged by water in fuel

Examination of the data in Table 8 shows that the overall particulate absorbance values for the samples filtered by D 2276 are higher for corresponding samples than those for the samples filtered using the field filtering device. This could be that the D 2276 filters were rated by the EOP while dry whereas the field filtering device filters were rated while still wet with fuel. Also, if the field filtering device allows fuel to flow around and between the filters, the results would be lower in value than expected. Most likely the explanation is a combination of the above two reasons. Note also that the particulate weights for corresponding samples tend to be higher for the D 2276 filters. This is probably also a result of the fuel flowing around and between the filters. It was also noted that color bodies present in the fuel tended to absorb onto the cellulose acetate-cellulose nitrate filter medium. These color bodies could absorb light but would not register as increased weight, thus leading to erroneous results.

Since the accuracy of the absorbance data obtained from the field filtering device filters was questionable, it was decided to use only the D 2276 filters to calibrate the EOP. Figure 4 is a plot of the particulate absorbance versus the particulate

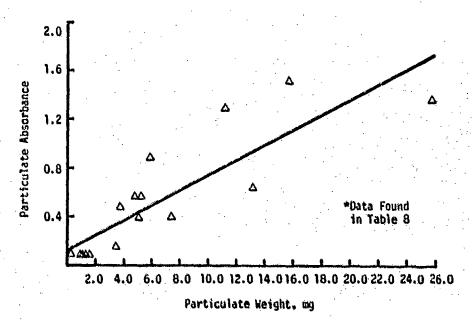


FIGURE 4. PLOT OF PARTICULATE ABSORBANCE VERSUS PARTICULATE WEIGHT DATA FROM TABLE 8 (Samples filtered using ASTM Method D 2276, mod., 1.2 μm)

weight for the D 2276 filter data from Table 8. Examination of the data shows, with few exceptions, an increase in particulate absorbance as particulate weight increases. For this reason, it was felt that the EOP was a viable approach to estimating the amount of particulates on a filter.

To measure color, the Hellige oil comparator device was chosen because it is small, portable, easy to use, and requires no light source other than the sun. Color evaluations were run both by ASTM Method D 1500 and the oil comparator. As shown in the results of these color evaluations (Table 9), the oil comparator gave results equivalent to D 1500. Although the values for D 1500 sometimes varied from the oil comparator values by 0.5 unit, this slight deviation is within the precision of the D 1500 method. The color evaluation is normally run as an adjunct to other stability tests as described in Appendix A. Procedures for the color test are given in Appendix B.

TABLE 9. DETERMINATION OF COLOR BY ASTM METHOD D 1500 VERSUS THE HELLIGE OIL COMPARATOR

Sample Code No.		6574	6624	6625	6626	6630	6638	6778	6716	6746
Next Fuel					**					
D 1500		1.0	3.0	1.0	2.0	6.0	0.5	4.0	3.0	0.5
Oil Comparator		1.5	3.5	1.0	2.5	6.5	0.5	4.0	3.0	0.5
D 2274 Filtrate	• • •	· .		* 51 1					:	
D 1500		4.0	3.0	1.5	3.0	6.5	0.5	4.5	2.5	1.0
Oil Comparator		4.0	3.5	1.5	3.0	6.5	0.5	4.0	2.5	0.5
150°C (3 hr Aging), Fili field device filtration	rate from									
D 1500	J. 1997	6.5	4.0	1.5	4.0	7.0	0.5	5.0	4.5	1.0
Oil Comparator		7.0	4.0	2.0	4.0	7.0	0.5	5.0	5.0	1.0

See Appendix B.

The filterability ratio test for cleanliness was performed on four of the fuels. This test measured the time required to filter a given amount of contaminated fuel and then compared this to the filtering time for an equal portion of the same fuel that

has been prefiltered. A value of 1.00 for the test indicates a perfectly clean fuel. Table 10 contains the results of this test. This procedure could very well be used in conjunction with the EOP to evaluate fuel cleanliness. However, this test requires a constant vacuum source which would eliminate the use of a hand pump. It may be possible to perform this test in a field environment with over-pressure on the input side of the filter being used to create a differential pressure across the filter, rather than using vacuum on the output side to create the differential. The low volume and low pressures required could be supplied from a small tank of compressed air or inert gas through a suitable constant pressure regulator. However, this approach has not as yet been tested.

TABLE 10. RESULTS OF FILTERABILITY RATIO TEST AND FILTRATION BY ASTM METHOD D 2276

Cample Code No.	6624	6625	6625	6630
Sample Code No.	0024	0023	0040	0030
Filterability Ratio, 1.2 µm.				
500 ml samples	28.30	 1.32	1.22	1.67
Particulates, D 2276 mod.,	· · · · · · .			
1.2 µm, mg/500 ml	13.10	1.00	1.36	15.58

To estimate the stability of the fuel, samples were heated at 150°C for 3 hours. Table 11 contains the data from this aging procedure. Included in the table are the particulate weights from both the field filtering device filtration and the D 2276 filtration. Also included in Table 11 are the results of the ASTM Method D 2274 test of the neat fuel.

After examining the data, it was judged that a linear relationship did not exist between the 150°C test data and the D 2274 test data. However, a go/no-go value for the 150°C test might be determined. It was also decided to shorten the 3.0-hour aging period to 1.5 hours. Not only was 3 hours considered too long for a field test, but it was also throught to be more extreme than necessary.

TABLE 11. COMPARISON OF 150°C TEST DATA TO DATA FROM ASTM METHOD D 2274)

D 2274, Sample Code Total Insolubles,		150°C Test, (3.0 hr aging) Particulate Weight, mg/100 ml, 1.2 μm			
No	me/100 ml	Field Filtering Device	D 2276		
6638	0.1	. 0.1	0.1		
6626	0.2	0.9	1.1		
6746	0.3	1.4	1.1		
6624	0.6	5.3	5,3		
6678	1.0	3.7	4,5		
6625	2.0	- 2.7	3.1		
6630	2.1	7,8	10.0		
6574	7.4	12.5	6.7		

The data generated during the evaluation of the laboratory prototype indicated that the approach to measuring the stability and cleanliness of fuel was a viable one. However, some areas needing improvements were identified.

- (1) All the electrical instruments in the prototype device were dependent upon 120 volts alternating current (VAC) for power. This dependence upon line current was a limiting factor to in-field use.
- (2) A stable power supply for both the lamp and the detector was needed.

 Conversion of existing circuitry or locating a lamp and detector that could be powered by one or two 12-volt batteries were possible solutions.
- (3) Color bodies from the fuel tended to adsorb onto the membrane filters. If water was in the fuel, the sample would filter more slowly through the membrane filters or possibly not filter at all.
- (4) The filter holder needed improvement so that fuel could not flow around the edge of the filters.

IV. PORTABLE FIELD FUEL QUALITY MONITOR

The development of the first prototype of the Field Fuel Quality Monitor (FFQM) is discussed in an earlier report (6) and Section III of this report. This section describes and discusses the test and evaluation of the most recent version of the FFQM.

A. Description of the FFQM

The FFQM is contained in two carrying cases. The case containing the CFMD, the sampling equipment, and the filtering equipment is 18 in. x 26 in. x 9 in. Fully loaded, this case weighs 30 pounds. The second case contains the 150°C test equipment. Fully loaded, this 10 in. x 16 in. x 11.5 in. case weighs 25 pounds. The two cases and equipment are pictured in Figure 5.

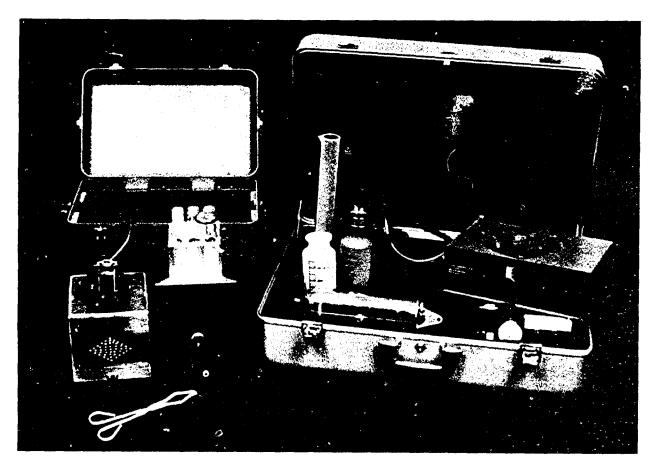


FIGURE 5. THE FIELD FUEL QUALITY MONITOR

Reference is once again made to the flow chart in Figure 1. In order to sample the fuel from its storage container (can, vehicle fuel cell, etc.), an apparatus such as the one pictured in Figure 6 is used. A vacuum is applied to the sample container by using the hand pump, and the fuel is drawn into the can through the tubing.

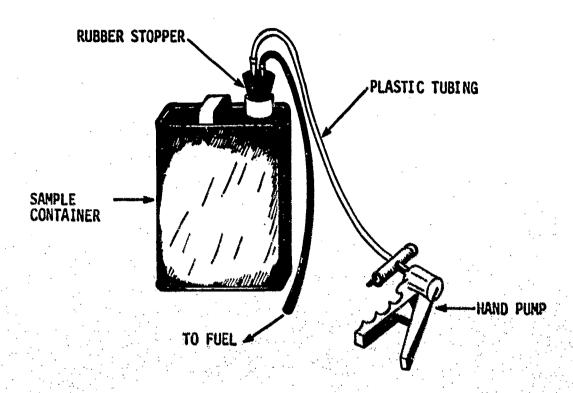


FIGURE 6. FUEL SAMPLING APPARATUS

To filter the fuel, the apparatus pictured in Figure 7 is used. This filtering apparatus was used in preference to the device used for filtering in the laboratory prototype. The filter holder in the newer apparatus does not allow fuel to flow around the edge of the filters as did the polycarbonate filter holder. A 500-mL aliquot is taken from the fuel sample and filtered through two glass fiber filters. Glass fiber filters are used because color bodies may adsorb onto the membrane filters which could alter the readings. It is important to note that the glass fiber filters must have a nominal porosity of 1.5 micrometers. These two filters (a sample filter and a control filter) are rated for amount of particulate matter present using the Contaminated Filter Measuring Device (CFMD) pictured in Figure 3. The CFMD is essentially a field-portable model of the EOP described earlier. The CFMD rates a filter by beaming light through it and measuring the amount of light that passes through. The readings are compared to a calibration curve to estimate the amount of particulate matter present.

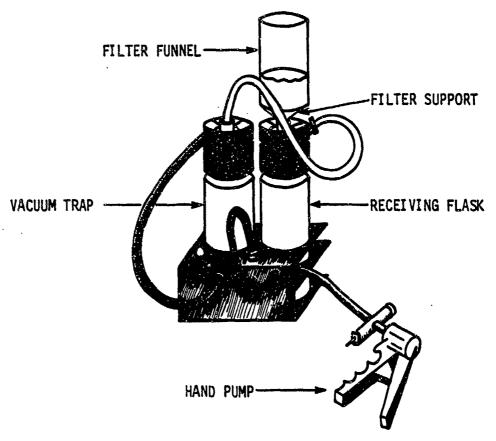


FIGURE 7. FUEL FILTERING APPARATUS

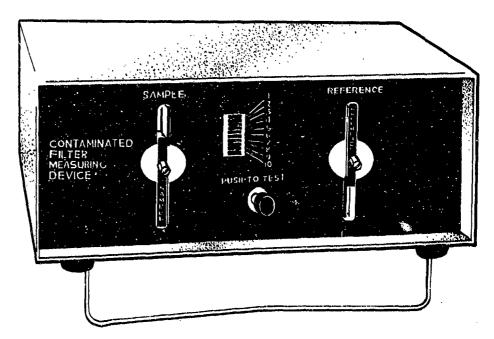


FIGURE 8. CONTAMINATED FILTER MEASURING DEVICE

The color comparator described in the preceding section was used to measure the color of the fuel. This device is small, portable, and does not require a special light source. The device gives results that are comparable to results from ASTM Method D 1500.

To age the fuel, 50-mL fuel samples are prefiltered, placed in test tubes, and heated to 150°C in a small, 115 VAC heating block as pictured in Figure 9. This aged fuel is then filtered using glass fiber filters, and the filters are rated using the CFMD.

B. Detailed Description of the FFQM

Figure 10 is a block diagram for the CFMD. Figure 11 is the wiring schematic for the CFMD, and Figure 12 is the layout pattern for the printed circuit board in the CFMD. To measure the amount of particulate matter in a fuel, a sample of the fuel is filtered through two sandwiched filters (a sample and a control filter). The filters are then placed in the appropriate test chamber in the CFMD. The amount of light which passes through each filter is detected by the photocell. The output from each of the photocells is amplified and sent to a voltage comparator. The voltage comparator ratios the two signals. The resultant signal is fed through a string of controlling resistors to a light emitting diode (LED) bar graph indicator. The LED indicator is a series of ten LED's with the strength of the signal determining the number of lights which come on. A strong signal, which results from a relatively clean sample filter, will illuminate more LED's than a weak signal (dirty sample filter).

The value of each resistor in the string was calculated so that the approximate particulate weight range for each LED is as indicated in Table 12. These levels are set by measuring the actual output for a series of filters. The desired weight ranges were selected, and the value of each resistor in the string of controlling resistors was calculated so that the appropriate number of LED's will illuminate based on the output signal. Table 13 provides the values for each of the resistors in the string. Figure 13 is a plot of CFMD results against actual weight of particulate on the filter to illustrate this relationship. In a recent program sponsored by the U.S. Navy (7), the CFMD was compared to two other methods for estimating the amount of particulate on a filter. After the filters were tested in

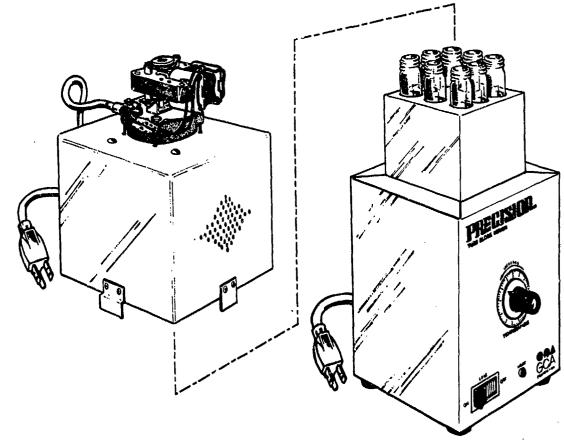


FIGURE 9. FUEL HEATING BLOCK

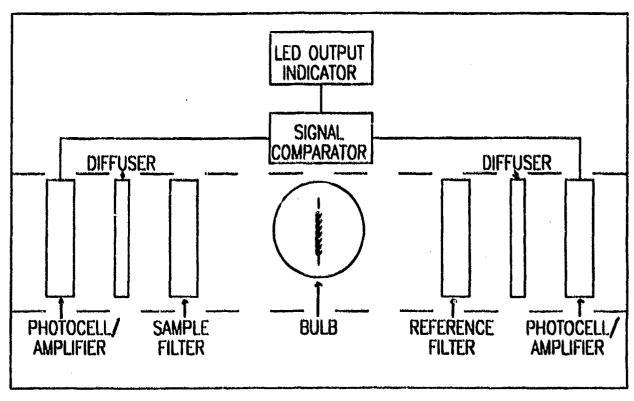


FIGURE 10. BLOCK DIAGRAM FOR CONTAMINATED FILTER MEASURING DEVICE

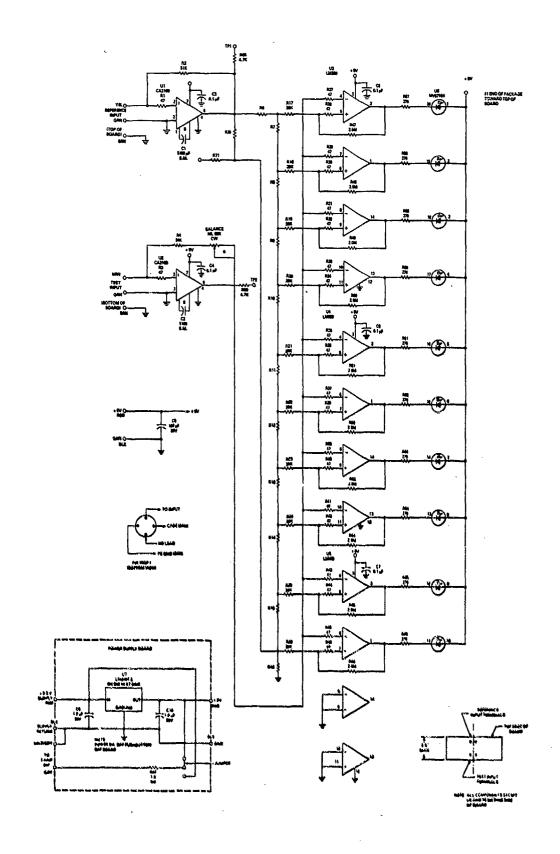


FIGURE 11. WIRING SCHEMATIC FOR CONTAMINATED FILTER MEASURING DEVICE

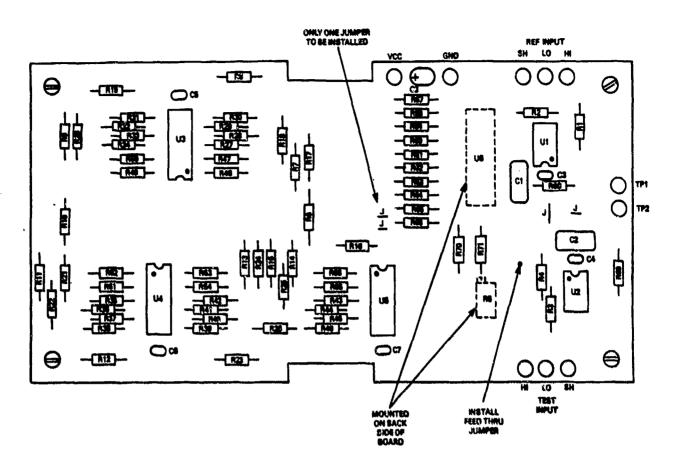


FIGURE 12. LAYOUT FOR PRINTED CIRCUIT BOARD FOR THE CONTAMINATED FILTER MEASURING DEVICE

TABLE 12. RELATIONSHIP OF CFMD LAMPS ILLUMINATED TO WEIGHT OF PARTICULATE ON TEST FILTER

No. of Lamps Illuminated	Approximate Weight Range of Particulate on Filter, mg		
10	Sint	0-1.5	
9	•	1.5-2	
8	-	2-3	
7	•	3-4	
6	-	4-5	
5	104	5-6	
4	•	6-7	
3	***	7-8	
2	-	8-10	
1	•	>10	

TABLE 13. RESISTOR VALUES FOR THE CFMD CONTROLLING RESISTOR STRING

Controlling Resistor	Calculated Value	Actual Value Ω
R7	1.10 K	1.10K
R8	770	787
R9	440	442
R10	220	215
R11	330	332
R12	220	215
R13	110	110
R14	220	215
R15	110	110
R16	0	Jumper
R6	7.48K	7.50K
R70	∞	Open
R71	 11,000K	Open .

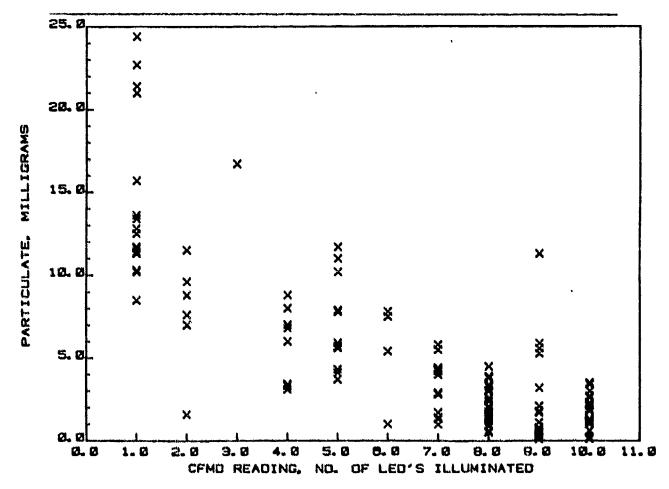


FIGURE 13. RESULTS FROM CFMD VERSUS WEIGHT OF PARTICULATE ON THE FILTER

the CFMD, they were rinsed with heptane to remove the fuel, oven dried, and reweighed to determine the weight of nonvolatile particulates collected. The dry filters (sample filter only) were then rated using two different visual comparison charts (DuPont Method F-21 and ASTM Method D 2276, Appendix C). These data are presented in Table 14. The CFMD, DuPont, and ASTM data are also plotted in Figure 14. The plots of CFMD data in Figures 14 and 15 are slightly different. This is attributed to the fact that the data in Figure 14 are from a smaller set of samples. Examination of these plots shows that the CFMD data exhibit good sensitivity throughout the range tested. The DuPont and ASTM data have very little sensitivity, especially above 5 milligrams of sediment.

Note that these CFMD results are reported in percent transmittance (%T). The CFMD is designed so that the operator may determine a more precise answer using a voltmeter. Test points are provided on the back of the unit to which a voltmeter

TABLE 14. FILTER RATING BY VARIOUS RATING METHODS

Gravimetric Particulate Weight, D 2276,mg	Navy Diesel Fuel Sediment Tester, Scale Reading	Army CFMD*, % T	duPont† Chart Rating	ASTM‡, Chart Rating
0.4	0.8	69	8	2
0.9	0.8	68	9	3
1.8	3.0	53	18	9
2.5	3.0	63	20	9
2.4	3.6	56	17	6
3.0	3.4	46	16	6
3.0	4.0	51	20	7
3.4	3.8	64	16	6
3.7	4.4	43	20	9
4.0	4.6	38	18	7
4.5	4.4	39	18	7
5.7	4.6	37	20	9
6.0	6.8	25	20	10
7.5	6.8	6	19	10
8.2	6.2	32	20	9
9.4	6.8	13	20	10
10.3	6.8	8	20	10
10.7	6.8	13	20	10
12.2	6.8	4	20	10

^{*}Army's Contaminated Filter Measuring Device †duPont F-21 Test Reference Blotters

[#]ASTM D 2276 Color Standards, Appendix A3

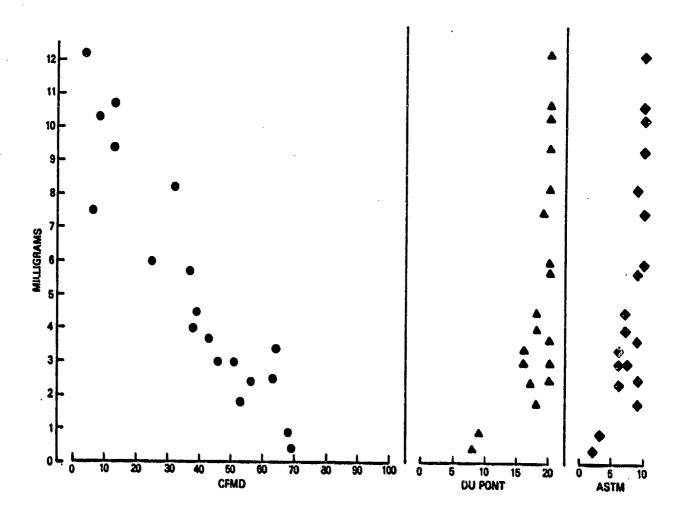
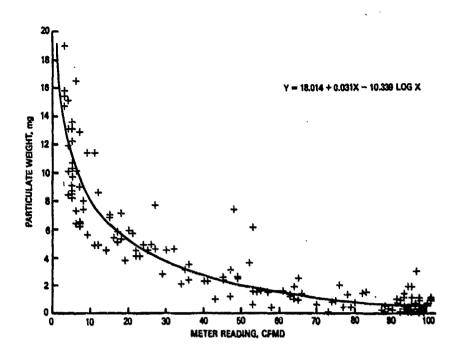


FIGURE 14. PLOT OF FILTER RATINGS VERSUS SEDIMENT WEIGHT

may be attached. The voltmeter readings from each channel (sample and reference) are ratioed to determine the %T. This %T value can then be converted to the approximate milligrams of sediment on the filter using a calibration chart (see Figure 15).

It should be noted that the calibration chart was developed using mostly fuels that contained brown colored particulates. When the particulates are black in color, false reading may be obtained with the CFMD. Black particulates tend to absorb more light than brown particulates and thereby give a false high reading. Some examples of false results obtained with black particulates are given in Table 15. One possible way to eliminate this problem is to find a light source and detector at



CALIBRATION CHART

<u>%T</u>	mg	%T_	mg	NT_	mg	<u>%T</u>	mo_
0							
1	18.0	26	4.2	51	1.9	76	.9
2	15.0	27	4.0	62	1.9	77	.9
3	13.2	28	3.9	63	1.8	76	
4	12.0	29	3,8	54	1,0	79	.3
5	11.0	30	3.7	56	1.7	80	.3
8	10.1	31	3.6	54	1.7	81	.\$
7	9.5	31 32 34 36	3.4	67	1.6	82	.a .a .7 .7 .7 .7 .8 .6 .8
8	9.0	33	3.3	58	1.6	83	.7
9	8.4	34	3.2	5 8	1.5	84	.7
10	8.6	36	3.1	60	1.6	86	.7
11	7.6	36 37	3.0	61	1.4	86	
12	7.2	37	2.9	62	1.4	17	.6
13	. 6.9	38	2.8	63	1.3	**	.0
14	6.6	39	2.8	64	1.3	80	.8.
18	6.3	40	2.7	66	1.3	10	.8.
18	6.1	41	2.6	98	1.2	91	.8
18 17	5.0	42	2.6	47	1.2	12	.5
18	5.6	43	2.4	**	1.2	13	.5
19	5.4	44	2.4	*	1.1	94	.Š
20	5.2	46	2.3	70	1,1	96	.5
21	5.0	48	2.2	71	1,0	98	.8
22	4.0	47	2.1	72	1.0	97	.8 .8 .8 .8 .8 .8 .8 .8 .8 .4
23	4.6	48	2.1	73	1.0	90	Ä
23 24	4.5	49	2.0	74	1.0	90	.4
25	4.3	50	20	76	1.0	100	Ä

FIGURE 15. CALIBRATION CHART FOR THE CFMD

TABLE 15. EXAMPLES OF FALSE CFMD RESULTS OBTAINED WITH BLACK PARTICULATES

No. of LED's Illuminated	Particulate Weight by CFMD, mg	Actual Gravimetric Weight, mg*
4	6-7	1.6
3	7-8	1.4
2	8-10	1.4
4	6-7	1.6
2	8-10	1.5 .
1	>10	1.6
1	>10	1.9
1	>10	2.2
1	>10	2.1
4	6-7	1.3
1	>10	1.9
1	>10	1.8
1	>10	2,1
1	>10	1.9
i	>10	1.8
1	>10	2.0

^{*}Determined using an analytical balance

a wavelength where neither black or brown particulate have spectroscopic absorptions.

The FFQM provides a means of estimating the stability of a fuel sample. A 50-mL sample of the fuel is aged at 150°C and then filtered. The test filters are rated by the CFMD to estimate the amount of material formed during aging. This test is most useful when used to monitor a given fuel supply over a period of time. A sudden rise in the results of the test could indicate a decrease in the stability of the fuel. The test is also useful for assessing the response of a fuel to stabilizer additives (such as MIL-S-53021). An attempt was made to determine the correlation, if any, between this test and the ASTM Method D 2274 (the accelerated stability test specified in the Army Diesel fuel specification, VV-F-800C). The 150°C test is a rapid test in which the test fuel is aged at 150°C, usually for either 1.5 or 3.0 hours. In ASTM D 2274, the fuel is aged for 16 hours at 95°C. Oxygen is bubbled through the fuel during aging in the D 2274 test; whereas, nothing is bubbled through the fuel during the 150°C test aging period. The amount of adherent insoluble material (gum) that is formed during aging is also determined in the D 2274 test. When the 150°C test is performed in the laboratory, the

amount of adherent insolubles formed may be determined. In a field test, however, it is not possible to make this determination due to lack of required equipment/materials. For this reason, the relationship between the 140°C field test and the D 2274 test is probably less than it could be.

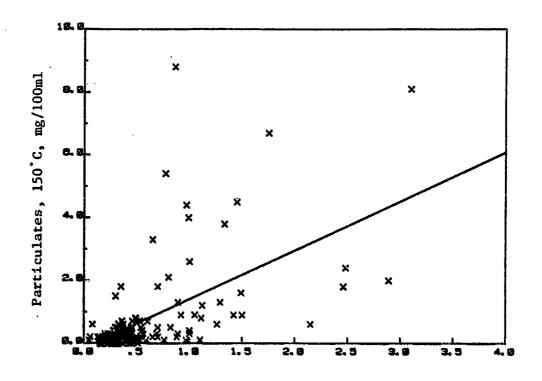
To determine this relationship, approximately 180 to 190 samples were analyzed by ASTM Method D 2274 and four versions of the 150°C test were evaluated using both the CFMD and an analytical balance. The gravimetric data were used for data analyses. The four versions of the test were as follows:

- 1) Age the fuel for 90 minutes; filter the aged fuel using a 1.5-micrometer pore size glass fiber filter
- 2) Age the fuel for 180 minutes; filter the aged fuel using a 1.5-micrometer pore size glass fiber filter
- 3) Age the fuel for 90 minutes; filter the aged fuel using a 0.7-micrometer pore size glass fiber filter
- 4) Age the fuel for 180 minutes; filter the aged fuel using a 0.7-micrometer pore size glass fiber filter

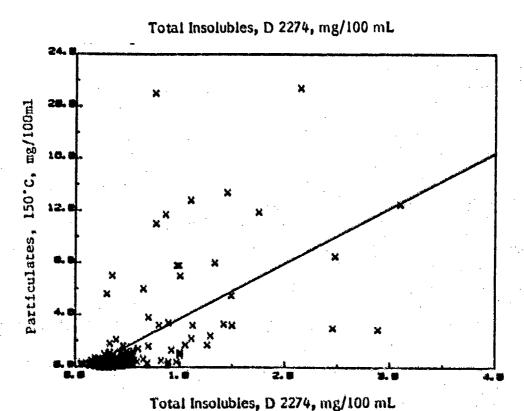
The data from these four versions were plotted against the total insolubles results for the ASTM Method D 2274. These plots are given in Figures 16 and 17.

In a field test such as this, it is not possible to determine the amount of adherent insolubles formed during the 150°C aging. Thus, the results of all four vers as of the 150°C test were plotted against the D 2274 filterable insolubles only, for comparison purposes. These plots are provided in Figures 18 and 19. A least squares, first degree polynomial regression was also performed on each set of data. The best straight line is drawn in each plot and the slope, intercept, and correlation coefficient are given in Table 16. Note that all the correlation coefficients are low indicating poor correlation, and none of the data has a significantly better correlation than the others.

It was also felt that many of the particles formed in the D 2274 test might be passing through the standard 1.5- μ m pore size glass fiber filter used in the method. For this reason, several of the D 2274 test samples were filtered a second time. The second filtration was through a 0.7- μ m pore size glass fiber filter. The

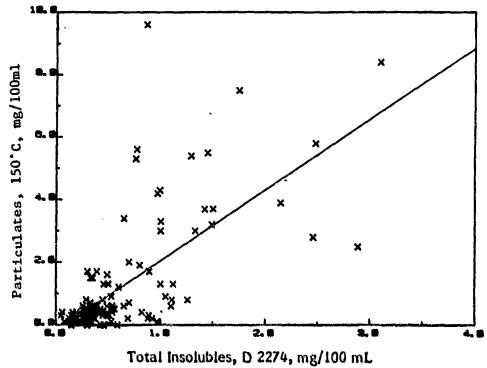


A. 90 Minutes

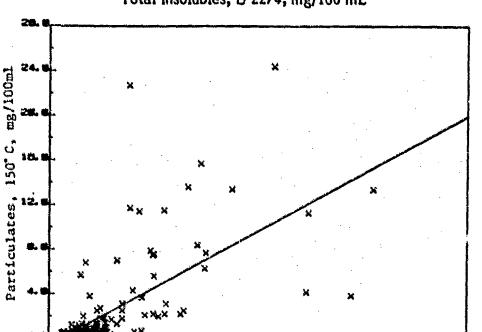


B. 180 Minutes

FIGURE 16. PLOT OF STANDARD ASTM METHOD D 2274
TEST RESULTS VERSUS 150°C TEST RESULTS AT
90 MINUTES AND 180 MINUTES AGING TIME
(150°C Test Aged Fuel was Filtered Through
a 1.5 µm Pore Size Glass Fiber Filter)



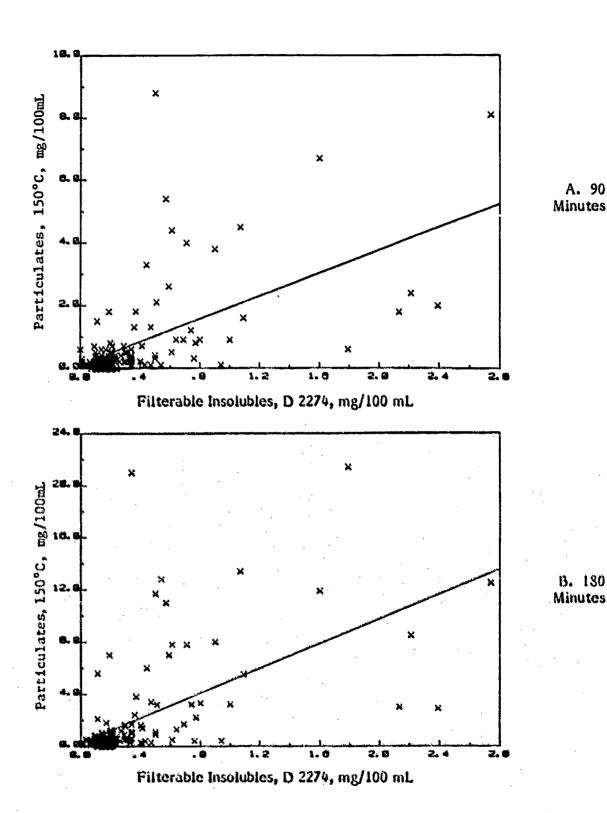
A. 90 Minutes



B. 180 Minutes

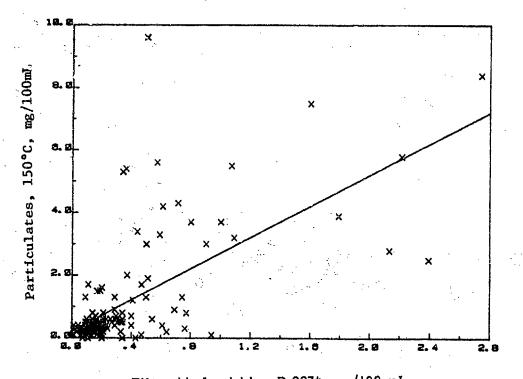
Total Insolubles, D 2274, mg/100 mL

FIGURE 17. PLOT OF STANDARD ASTM METHOD D 2274
TEST RESULTS VERSUS 150°C TEST RESULTS AT
90 MINUTES AND 180 MINUTES AGING TIME
(150°C Test Aged Fuel was Filtered Through
a 0.7 µm Pore Size Glass Fiber Filter)

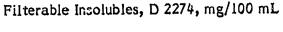


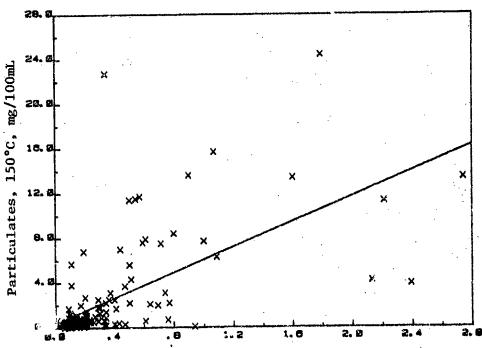
A. 90

FIGURE 18. PLOT OF ASTM METHOD D 2274 FILTERABLE INSOLUBLES **VERSUS 150°C TEST RESULTS AT 90 MINUTES AND** 180 MINUTES AGING TIME (150°C Test Aged Fuel was Filtered Through a 1.5 µm Pore Size Glass Fiber Filter)



A. 90 Minutes





B. 180 Minutes

Filterable Insolubles, D 2274, mg/100 mL

FIGURE 19. PLOT OF ASTM METHOD D 2274 FILTERABLE INSOLUBLES VERSUS 150°C TEST RESULTS AT 90 MINUTES AND 180 MINUTES AGING TIME

(150°C Test Aged Fuel was Filtered Through a 0.7 µm Pore Size Glass Fiber Filter)

TABLE 16. SLOPE, INTERCEPT, AND CORRELATION COEFFICIENT FOR THE LEAST SQUARES, BEST FIT LINES IN FIGURES 15-18

Figure	 -	•	Correlation
No.	Slope	Intercept	Coefficient (R ²)*
16 A	1.561	-0.155	0.332
16B	4.234	-0.506	0.359
17A	2,264	-0.217	0.484
17B	5.140	-0.606	0.422
18A	1.839	0.103	0.342
18B	4,741	0.283	0.334
19A	2,498	0.218	0.437
19B	5.686	0.376	0.382

^{*}R² of 1.000 = perfect correlation

particle weight obtained from the second filtration (0.7- μ m) was then added to the particle weight from the first (1.5- μ m) filtration. Figures 20-23 are analogous to Figures 16-19 using the revised filterable insolubles values. The slope, intercept, and correlation coefficient for each of the best fit lines in the figures are given in Table 17. Note that the correlation coefficients for Figures 20-23 are also quite low.

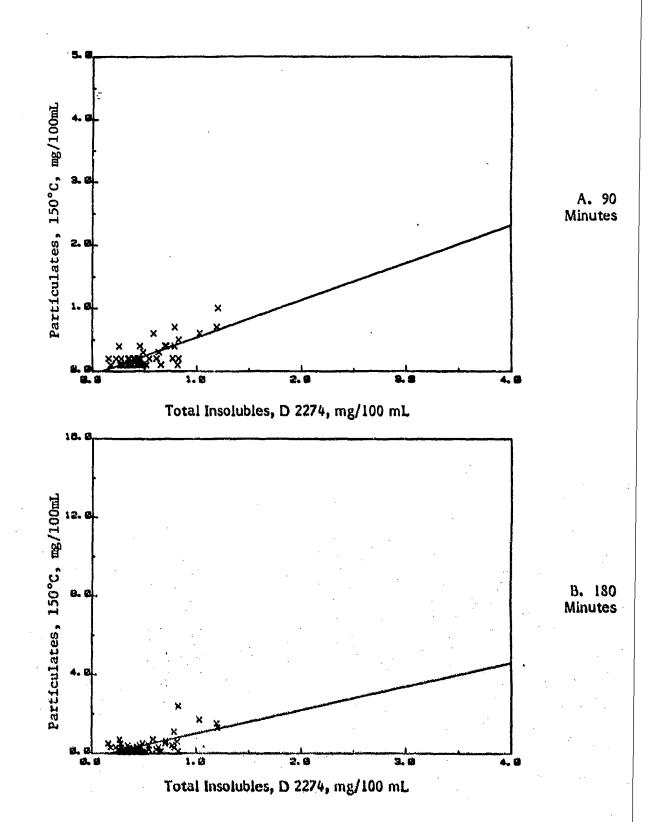
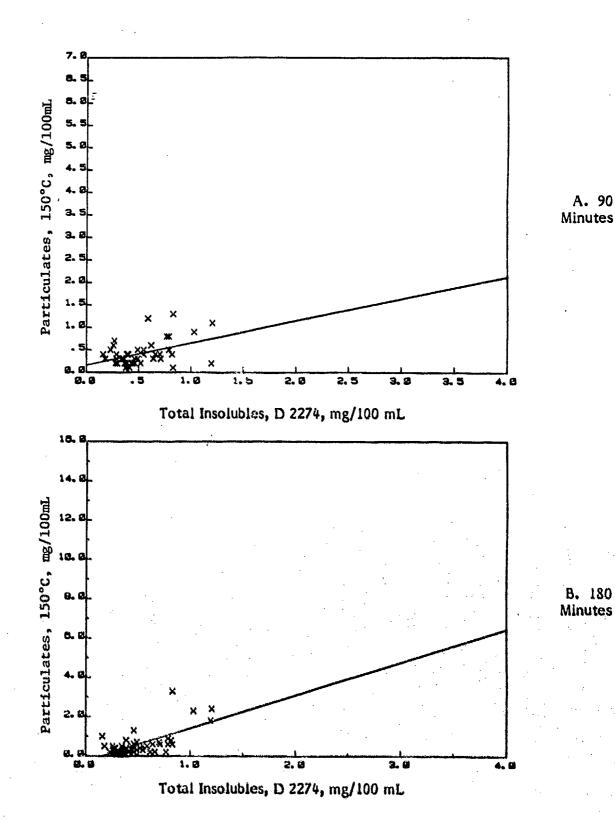
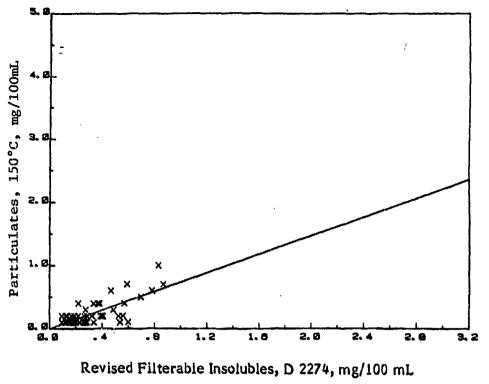


FIGURE 20. PLOT OF ASTM METHOD D 2274 TEST RESULTS, WITH REVISED FILTERABLE INSOLUBLES DATA, VERSUS 150°C TEST RESULTS AT 90 MINUTES AND 180 MINUTES AGING TIME (150°C Test Aged Fuel was Filtered Through a 1.5 μm Pore Size Glass Fiber Filter)



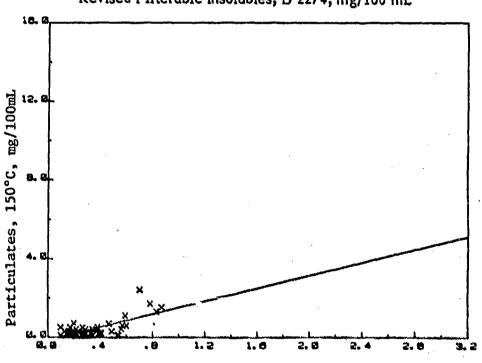
A. 90

FIGURE 21. PLOT OF ASTM METHOD D 2274 TEST RESULTS, WITH REVISED FILTERABLE INSOLUBLES DATA, VERSUS 150°C TEST RESULTS AT 90 MINUTES AND 180 MINUTES AGING TIME (150°C Test Aged Fuel was Filtered Through a 0.7 µm Pore Size Glass Fiber Filter)



A. 90 Minutes

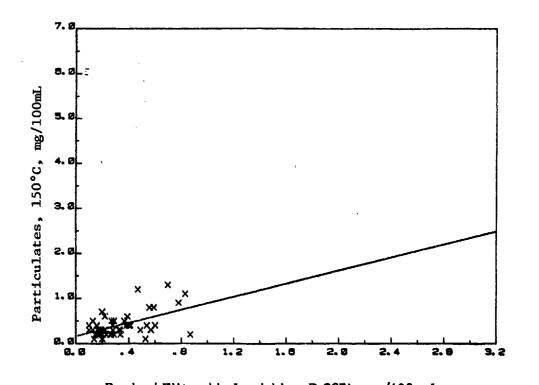
B. 180 Minutes



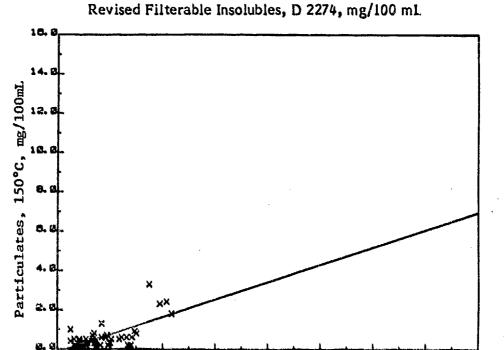
Revised Filterable Insolubles, D 2274, mg/100 mL

FIGURE 22. PLOT OF ASTM METHOD D 2274 REVISED FILTERABLE INSOLUBLES VERSUS 150°C TEST RESULTS AT 90 MINUTES AND 180 MINUTES AGING TIME

(150°C Test Aged Fuel was Filtered Through a 1.5 µm Pore Size Glass Fiber Filter)



A. 90 Minutes



B. 180 Minutes

Revised Filterable Insolubles, D 2274, mg/100 mL

FIGURE 23. PLOT OF ASTM METHOD D 2274 REVISED FILTERABLE INSOLUBLES VERSUS 150°C TEST RESULTS AT 90 MINUTES AND 180 MINUTES AGING TIME

(150°C Test Aged Fuel was Filtered Through a 1.5 µm Pore Size Glass Fiber Filter)

TABLE 17. SLOPE, INTERCEPT, AND CORRELATION COEFFICIENT FOR THE LEAST SQUARES, BEST FIT LINES IN FIGURES 19-22

Figure No.	Slope	Intercept	Correlation Coefficient (R ²)*
20A	0.595	-0.059	0.522
20B	1.198	-0.189	0.395
21A	0.492	0.161	0.188
21B	1,675	-0.253	0.406
22A	0.741	0.004	0.533
22B	1.640	-0.129	0.487
23A	0.729	0.167	0.272
23B	2.215	-0.143	0.467

^{*}R² of 1.000 = perfect correlation

V. FIELD TESTING AND EVALUATION

The FFQM underwent testing and evaluation at several Army installations including the POMCUS Fully Fueled Vehicle Test in Miseau, Germany; Fort Polk, LA; Alabama Army National Guard; Belvoir R&D Center, VA; Fort Carson, CO; Fort Hood, TX; and the 200th TAMMC, Zwiebrucken, Germany. An FFQM is still in use at each of the last three sites as of this writing. The purpose of this portion of the program was to:

- A) Demonstrate field fuel quality monitor utility/effectiveness,
- B) Identify any deficiencies and/or possible modifications,
- C) Provide initial assessment of user confidence in this equipment and suitability for use by troops, and
- D) Correlate on-site field test results with standard ASTM tests and fuel quality monitor results obtained under laboratory conditions.

Several user comments were received as a result of this phase of this program. Many of the comments dealt with particular changes to the users manual that would simplify use or operation. Examples include suggested illustrations to simplify the use of the equipment and suggestions for cleaning the apparatus. Some users had difficulty in adjusting the heating block to the proper temperature; however, a suitable replacement for this heater could not be found. All users were pleased with the size and ease of use of the CFMD. The user comments as a whole provided the type of information needed to make the FFQM useful and convenient to use by field personnel.

VL. CONCLUSIONS AND RECOMMENDATIONS

- A portable unit to measure the quality of distillate fuel in the field was developed.
- The feasibility of using light transmittance through a filter to measure the amount of contaminant present on a filter and thereby measure the cleanliness of a distillate fuel sample was shown. Light transmittance and gravimetric analysis were correlated for particulates on test filters. It was determined that initial lamp intensity of the Contaminated Filter Measuring Device (CFMD) had essentially no effect on the measured transmittance through the filter.
- The Field Fuel Quality Monitor underwent user testing and evaluation at several Army installations which provided much useful information for improving the FFQM.
- The 150°C test for fuel stability was judged to be most useful as a monitoring tool. The test was found to have poor correlation to ASTM Method D 2274.
- Black particulates may give false readings in the CFMD. Possible methods
 including light sources at other wavelengths, for reducing or eliminating this
 potential error should be investigated; including light sources at other
 wavelengths.

VIL REFERENCES

- 1. Stavinoha, L.L., Westbrook, S.R., and LePera, M.E., "Army Experience and Requirements for Stability and Cleanliness of Diesel Fuel," Interim Report No. AFLRL 128, U.S. Army Fuels and Lubricants Research Laboratory, Southwest Research Institute, San Antonio, TX, Government Accession No. AD A088008, July 1980.
- 2. Commercially available from Gulton Industries, Inc., Hawthorne, CA, and Telectro-Mek, Inc., Fort Wayne, IN.
- 3. Memorandum from J. H. Frazar, U.S. Army Fuels and Lubricants Research Laboratory, to file 10-5070-140, with the subject, "Field Test Kit for Diesel Fuel Contamination," 5 March 1979.
- 4. Described in "Operation and Maintenance Manual for Test Kit, Fuel Contamination," prepared for Headquarters U.S. Army Troop Support Command by Potomac Research, Inc., December 1974. Note: Not an official DA Publication and not available through AG publication channels.
- 5. Stavinoha, L.L., and Westbrook, S.R., "Accelerated Stability Test Techniques for Middle Distillate Fuels," presented at the American Society for Testing and Materials (ASTM) Symposium on Distillate Fuel Stability and Cleanliness, Chicago, IL, Special Technical Publication, No. 751, 24 June 1980.
- 6. Westbrook, S.R., Stavinoha, L.L., Barbee, J.G., and Mengenhauser, J.V., "A Field Monitor for the Stability and Cleanliness of Distillate Fuel," Interim Report AFLRL No. 137, U.S. Army Fuels and Lubricants Research Laboratory, Southwest Research Institute, San Antonio, TX, Government Accession No. AD A110694, December 1981.

APPENDIX A EXPERIMENTAL TEST PROCEDURES FOR DISTILLATE FUELS

APPENDIX A EXPERIMENTAL TEST PROCEDURES FOR DISTILLATE FUELS

The following experimental methods are used to test distillate fuels:

1. Color: ASTM D 1500-64

2. Steam Jet Gum: ASTM D 381-70 (Modified)

Modification: The residue is dried an extra 0.5 hr in the gum block if it still appears wet after the first 0.5 hr.

3. Particulate Contamination: ASTM D 2276-7 (Modified)

Modification: •47-mm diameter filters are used instead of 37-mm diameter filters.

- •1.2-micrometer pore size filters are used instead of 0.8-micrometer pore size filters.
- n-heptane is used as the solvent.
- •The sample size may range from 100 to 1000 ml.
- 4. Accelerated Stability: ASTM D 2274-74.
- 5. NACE Standard TM-01-72 Steel Corrosion: Antirust properties of petroleum products pipeline cargoes test method.
- 6. 43°C Storage Test: This test method is used for studying the storage stability of distillate fuels under vented 43°C storage conditions.

The bottles used were made of borosilicate glass (Curtis Matheson Scientific, Inc., Catalog No. 037-59 (1977), are rectangular (the cross section is 6.35 cm x 9.53 cm), and are 27.31 cm in overall height. The volume of each bottle is 1 liter. The bottles have a tool-finished neck for increased strength, with an opening that takes a No. 6 rubber stopper. A 10.2-cm square, thin sheet of Teflon was wired securely around the neck to cover the opening. A 0.64-cm diameter hole was punched into the sheet for insertion of a bent piece of glass tubing to act as a vent.

The storage bottles were cleaned with alkaline laboratory cleaner and then rinsed with water. Each bottle was filled approximately half full with chromic acid cleaning solution and rolled in such a way that all of the inner surface came in contact with the acid solution. The bottle was allowed to stand for at least 1 hr before it was rinsed four times with deionized water. The bottles were allowed to drain and then dry overnight in an oven at 150°C.

The fuels to be tested were prefiltered through a 1.2-micrometer membrane filter (Millipore Corp., Bedford, MA, Catalog No. RA-WP-047-00). Then four samples (650 ml per sample) of each fuel under study were placed in the bottles and properly labeled. The perforated caps previously described were placed on the bottles.

One bottle from each series was retained for analysis at zero weeks storage. The control samples were stored in the dark under nitrogen at -1° to 4°C until analyzed. The remaining bottles were placed in the 43°C oven. One bottle of each fuel was removed after 12, 16, and 32 weeks of storage. The samples were carefully inspected for the appearance of visual sediment and were analyzed for color by ASTM Method D 1500. Filterability ratio (modified) and steam jet gum were also determined. Then 100 ml of each sample were filtered, using a modified D 2276 method, through 1.2-micrometer membrane filter (as described above). The filter was dried and weighed. The resulting particulates were reported as mg/100 ml.

7. 80°C Storage Test: In this test, ten 120-ml volume sample bottles were used for each fuel. The caps of these bottles were fitted with a Teflon liner, and each liner was pierced with a 0.65 cm diameter hole. A bent glass tube was placed in the hole to act as a vent. Each bottle contained 105 ml of sample.

One control sample of each fuel tested was set aside under nitrogen and in the dark. The other nine samples of each fuel were placed in a force-draft oven at 80°C. Of these samples, three of each fuel were removed from the oven after 24 hr, another three samples after 72 hr, and the final sample of fuel after 168 hr. The samples were tested for color (ASTM D 1500), light absorbance, steam jet gum (D 381), visual sediment, and particulates (D 2276, modified, 1.2-micrometer pore size filter).

8. 150°C Accelerated Fuel Oil Stability Test: In this test, three 55-ml samples of a fuel were measured into 2.5 cm x 20.0 cm culture tubes with screw caps. These tubes were Kimax Brand glass from Curtis Matheson Scientific, Inc., Catalog No. 225-870 (1977). The tubes containing the samples were submerged to the necks in a 150° \pm 1°C oil bath. The caps on the tubes were loosened slightly to allow for air expansion in the tube. The samples remained in the batch for 90 minutes.

Two of the samples were combined and analyzed for color (D 1500), light absorbance, and particulates (D 2276, modified, 1.2-micrometer pore size filter) while the third was analyzed for jet gum (D 381).

- 9. ASTM D 873-74 Oxidation Stability of Aviation Fuels (modified), often referred to as the potential residue method, measures the tendency of fuels to form gum and deposits under accelerated aging conditions. The modifications made to this test and used in these studies are as follows:
 - •The gum solvent was replaced with n-heptane.
 - •The sintered glass crucible was replaced with a 1.2-micrometer filter using double filter tare procedure as in ASTM D 2276.
 - •Items A (particulate), B (insoluble gum), and C (soluble gum) were all reported as milligrams per 100 ml of sample (mg/100 ml).
 - •The color (D 1500) was reported for the treated sample.
 - •The visible sediment was reported.
 - The treated sample was examined for "sour" or "oxidized linseed oil" odor and reported.
- 10. Light Absorbance; Spectrophotometric Analysis of Fuels: Light absorbance was measured on a UV-Visible Spectrophotometer.

The light absorbance was recorded over the wavelength scanning range of 650 to 400 nanometers (nm) at a scanning speed setting of 2 nm per second, and the light absorbance values were reported at 650, 575, 540, and 500 nm.

The instrument was "zeroed" without cells in the light path. Deionized water was placed in both cells and scanned over the 650- to 400-nm range to determine the cleanliness of the cells and any possible deviation of the baseline. The cells used were 1-cm corex cells which had been cleaned with solvents, detergents, and/or cleaning solution as required to maintain a zero baseline (0.005 absorbance units). The deionized water was removed from the sample cell, and the cell was rinsed with acctone and dried with clean air. The fuel to be examined was placed in the sample cell, and the scan was repeated. For each fuel examined, the cell was cleaned as noted previously. Periodically, the zero and baseline scan were checked with deionized water.

11. Method for Testing Fuel Filterability

•Discussion: In this method, 500-ml samples of prefiltered and neat test fuel were filtered through a 1.2-micrometer filter disc. Fuel filterability was evaluated by the ratio of the filtering time for the neat test fuel to the filtering time for the prefiltered fuel.

Equipment

- a. 1-092-10V1, Air Pump
- b. 1000-ml Filter Flask
- c. 500-rnl graduate cylinder
- d. Stop watch
- e. XX10-047-00 Pyrex Filter Holder
- f. 1.2-micrometer Filter Disc No. RAWP-047-00
- g. Petri-Dishes PD-10-047-00

Suggested Supplier

Fisher Scientific

Fisher Scientific

Fisher Scientific

Fisher Scientific

Millipore Corporation

Millipore Corporation

Millipore Corporation

APPENDIX B PROCEDURE FOR OPERATION OF FIELD FUEL QUALITY MONITOR

INTRODUCTION

The cleanliness of the fuel is evaluated by first measuring the fuel color, then filtering the fuel and determining the amount of particulates on the filters using a particulate-measuring device. The filters are rated by measuring the amount of light which passes through the filters to estimate the amount of particulates. A measure of the stability of the fuel is obtained by aging a sample of the fuel at 150°C for 1.5 hours and determining the amount of sediment formed.

OPERATING PROCEDURE

1. General Instructions.

WARNING

Handle the equipment, components materials, and fuels with utmost care. Dangers and hazards inherent in handling highly flammable liquid fuels require constant observance of and strict adherence to rules governing safety.

- 1.1 Be thoroughly familiar with the contents of the monitor and the uses and functions of each component before performing any test. When handling fuels, pay particular attention to the following safety precautions.
 - Provide adequate ventilation.
 - · Permit no smoking or open flames in the testing area.
 - Keep containers capped when filled with fuel.
 - Handle liquids carefully to avoid spills or splashes.
 - Immediately wipe up any liquid that has spilled, and discard wiping materials in a metal container.
- 1.2 Always have a chemical fire extinguisher in operating condition nearby, and know how to use it.

- 1.3 The following additional items are required for operation and maintenance of the monitor:
 - Clean rags and detergent for general cleanup of equipment.
- 1.4 The precautions required to assure a representative sample depend on the type of product being sampled, the source from which it is drawn, and the sampling procedure employed. A procedure suitable for the sampling conditions should be used. (Chapter 4 of MIL-HDBK-200F gives detailed instructions for sampling). A Bacon Bomb sampler is included as part of the monitor (Figure 1).

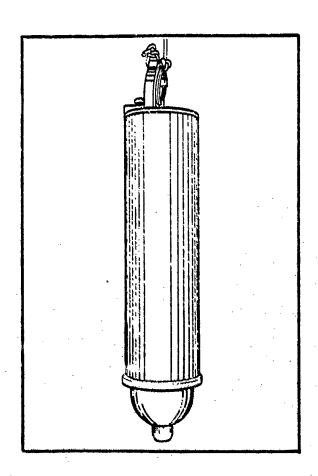


FIGURE 1. BACON BONE SAMPLER

1.5 Improperly taken samples can completely invalidate a test. Only responsible and trained personnel should be assigned to obtain sample prod-

ucts. The importance of obtaining representative samples cannot be overstressed because analysis will not give reliable data on a product if the sample is not:representative.

- 1.6 This manual will in no way alter any assigned responsibility of various activities for submitting special samples to a designated laboratory.
- 1.7 Do not take samples through storage tank cleanout lines, manifolds, water draw-offs, bleeder valves, hose nozzles, etc., as such samples will not be representative of the product in the tank. When it is necessary to sample service station tanks, and access to such tanks cannot be gained through a manhole or sampling hatch, the tanks may be sampled through the servicing hose after first discharging from the hose a volume of the product estimated at two times the volume contained in the piping system.
- 1.8 Identify each sample container immediately after sampling by securely attaching a sample tag. Information on the tag should include the location of the activity at which the sample is taken, name of person taking the sample, grade of material, quantity represented, specification of material when known, storage tank number and location, date sample was taken, type of sample, and reason for sample.
- 2. Determining the Color of the Fuel.
- 2.1 Color should be determined on the unfiltered fuel.
- 2.2 Open the front cover of the Comparator housing and place the center of the Color Disc on the ring with the number plates facing the operator. Close the cover. As the Disc is rotated, the number plates representing ASTM Color Numbers can be read through the upper opening on the right-hand side of the front cover. The alignment is such that only one figure is visible when a glass Color Standard is completely in the field of view.
- 2.3 The observation fields for colorimetric comparison can be seen through the two central openings. Through the prism attachment, they are seen as

half-fields. The right-hand field is produced by the test sample in a tube which is placed in the opening at the right of the instrument, while the left-hand field is formed by one of the glass Color Standards.

2.4 Fill a tube with the test sample to a height convenient for full exposure in the observation field and place it in the right-hand opening of the apparatus. Make the color comparison by revolving the Color Disc so that one Standard after another is brought into the observation field. When using the Comparator with the prism attachment, view the color fields with the eye in line with the center of the fields. The half-fields should be free of shadow effect which can be caused by off-center or oblique observation. For normal eyesight, the preferred viewing distance is approximately 10 inches.

NOTE

Do not make the mistake of placing the eye close to the Comparator housing. Do not prolong the observation for more than 10 to 15 seconds. For very accurate readings, let the eyes rest between such intervals, preferably by viewing a gray or green surface.

- 2.5 When a color match is obtained between the test sample and one of the glass Standards, the figure seen in the upper opening in the front cover gives the direct reading. If the color of the test solution is intermediate between those of two glass Standards, the result to be reported will be intermediate between their corresponding values and may be estimated by interpolation.
- 2.6 While making readings, the opal glass plate at the back of the Comparator shall face directly toward the light, and care should be taken that pronounced shadows do not cause uneven illumination of the Comparator fields. Northern exposure provides the best daylight for colorimetric determination, but any indirect light from outdoors usually will be satisfactory. Direct sunlight should always be avoided, even in the early morning or late afternoon.

- 3. Filtering the Fuel Sample.
- 3.1 Assemble the fuel filtering apparatus as pictured in Figure 2.

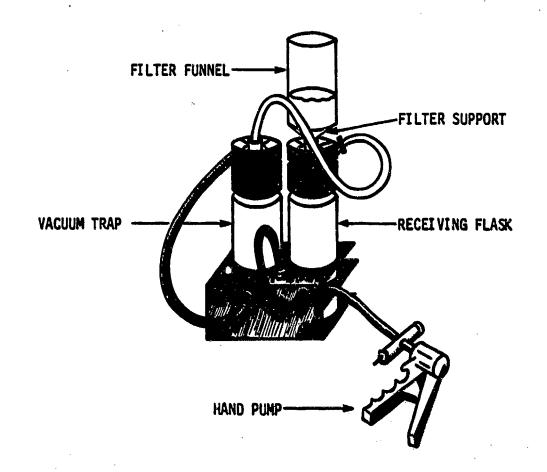


FIGURE 2. FUEL FILTRATION ASSEMBLY

- 3.2 Remove the filter funnel and, using the forceps, place two of the glass fiber filters onto the filter support. Note: Be certain the "rough" side of the filters is up.
- 3.3 Replace the filter funnel, making certain the two filters remain in place.
- 3.4 Shake the sample can and rinse the graduated cylinder with 50 mL of fuel to prevent carryover from the previous sample. Measure 250 mL of the fuel to be tested, apply vacuum to the flask with the hand pump, and pour the fuel into the filter funnel.

- 3.5 When all 250 mL of the fuel have been filtered, release the vacuum and remove the cap from the receiving flask. Do not separate the filter funnel from the filter support or otherwise disturb the filters.
- 3.6 Pour the filtered fuel into a suitable container for disposal.
- 3.7 Replace the receiving flask cap, shake the sample can, and measure another 250 mL of fuel sample.
- 3.8 Pour this sample into the filter funnel and apply vacuum.
- 3.9 When all of this 250 mL sample has been filtered, release the vacuum.
- 3.10 Remove the cap from the receiving flask and SAVE THE FILTERED FUEL in a suitable clean container, labeled with the sample number, for future stability testing.
- 3.11 A total of 500 mL of fuel has now been passed through the filters in preparation for particulate measuring.
- 4. Measuring Particulates Using the Contaminated Filter Measuring Device (CFMD). See Figure 3.
- 4.1 Remove the "sample" filter holder and the "reference" filter holder from the CFMD and place on table near the CFMD.
- 4.2 Lift up the metal lid of each filter holder and inspect glass to ensure it is clean.
- 4.3 Remove the filter funnel from the filter support of the apparatus that was used in paragraph 3.1 through 3.11, taking care not to disturb the deposits on the filter pad.
- 4.1 Using forceps, carefully remove the top filter which has trapped the particulate, and place it on the glass surface of the "sample" filter holder of the CFMD.

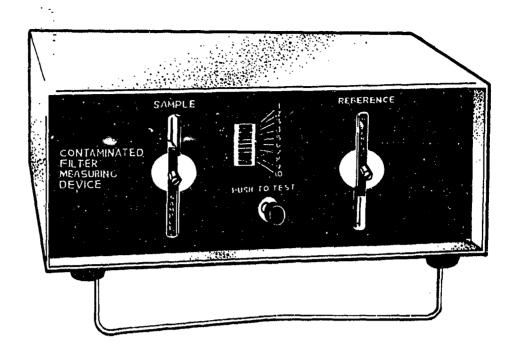


FIGURE 3. CONTAMINATED FILTER MEASURING DEVICE (CFMD)

- 4.5 Remove the bottom filter from the filtering apparatus and place it on the glass surface of the "reference" filter holder of the CFMD.
- 4.6 Close the metal lid of each CFMD filter holder, and wet each of the filters with 10 drops of clean filtered fuel.
- 4.7 Replace the filter holders in the slots of the CFMD, taking care to ensure that the proper holder is in each slot and that the holder was not inserted upside down. The identification labels on the edge of each holder should agree with the label above each slot.
- 4.8 Press the button marked "Push To Test" and record the number of lights which are illuminated on the LED bargraph readout. The number of lights

illuminated relates to the amount of particulate present on the sample filter as shown in Table 1.

TABLE 1. RELATIONSHIP OF CFMD LAMPS ILLUMINATED, TO WEIGHT OF PARTICULATE ON TEST FILTER

The state of the s

Number of Lamps Illuminated		Approximate Weight of Particulate on Filter in Milligrams
10	•	0-1.5
9	**	1.5-2
8		. 2–3
7	=	3–4
6	=	45
5	=	5-6
4	-	6-7
3		7-8
2	•	8-10
1	•	>10

4.9 Should the CFMD fail to operate, remove the battery compartment cover from the rear of the unit and replace the "C" size batteries (6 each). Be sure to observe proper polarity when replacing batteries. The CFMD is designed to use little power so the batteries should last at least 4 months in normal service.

Measuring for Stability.

5.1 Summary of Method: A measured volume of distillate fuel is aged 90 minutes at 150°C (302°F) in an open tube with air exposure. After aging and cooling, the fuel is filtered, and the amount of insoluble particulates that are formed is estimated.

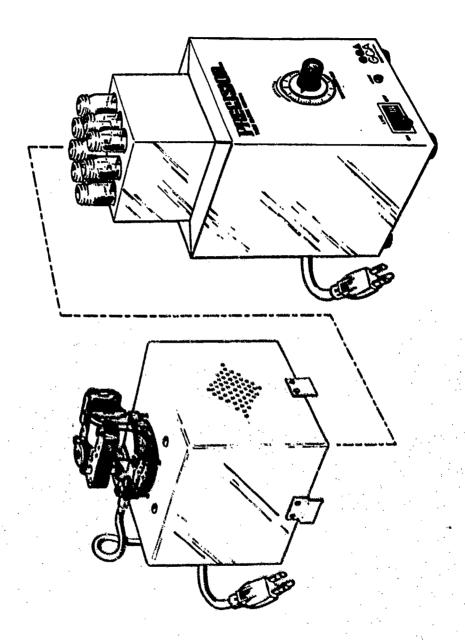
WARNING

There are exposed hot surfaces on the heating apparatus (see Figure 4), which can cause severe burns. Avoid contact with exposed skin by use of protective equipment as required. The oil sample vapors must be properly vented to prevent their entering the work atmosphere. The block should be located in an open area free of sparks and open flames.

- 5.2 Place the empty tube block into the heating base.
- 5.3 Place the thermometer into the tube block and set the blower support in place on the heating base. Do not put the blower onto the blower support at this time.
- 5.4 Plug in the heating base and adjust the heater control until the thermometer reads a stable 150°C for 15 to 20 minutes.
- 5.5 Using the graduated cylinder, measure 50 mL of the filtered fuel from the cleanliness testing procedure (paragraph 3.10 above).
- 5.6 Pour the 50-mL sample into one of the aging tubes.
- 5.7 Remove the thermometer from the tube block.
- 5.8 Place the uncapped sample tube(s) into the tube block.
- 5.9 Screw the charcoal filter package into the threads on the underside of the blower assembly. Set the blower in place, and plug in the blower.
- 5.10 Heat the sample(s) for 90 minutes ± 3 minutes.

WARNING

Fuels will be heated above their flash points. Fuels with initial boiling points below that of No. 2 fuel (kerosene, Jet A) may boil under the conditions of the



test. Fuel samples which may contain gasoline or other volatile materials may vaporize excessively and should not be tested. The block must be located in a ventilated area and kept away from sparks and open flame.

- 5.11 Remove the sample(s) from the block and allow them to cool gradually to 21° to 26°C (70° to 80°F) over a period of 1.5 to 4 hours in a location shielded from light. Do not allow the samples to cool more than 4 hours.
- 5.12 Prepare the filtration assembly with two new filters and filter the aged fuel sample. Note: If the deposit on the filter pad is not evenly distributed, the pad should be rejected, and the test rerun after cleaning the filtration apparatus.
- 5.13 Determine the amount of particulate present on the filters as described in Section 4 above.
- 5.14 When tube block has cooled to room temperature, clean the block with a warm detergent solution. Also clean the aging tubes in solvent* and a warm detergent solution. The heating base should be cleaned by wiping it with a clean, dry rag.

^{*} Possible solvents include paint remover, carburetor cleaner, and TAN (a mixture of equal parts toluene, acetone, and methanol). This solvent rinse is to remove gum deposits from the walls of the aging tube.

APPENDIX C

PARTS LIST AND LIST OF MANUFACTURERS AND SUPPLIERS

PARTS LIST

Part No.	Description	Manufacturer/ Supplier	Qty Inc. in Unit
AL-001	Contaminated Filter Meas. Device	001	l ea
110X	Carrying Case (15"x26"x9")	002	l ea
ZIP 850	Carrying Case (10"x16"x11.5")	002	l ea
149 4954	Filter, Neutral Density	003	2 ea
6238	Thermometer, 0°-250°C Range	Ö 10	l ea
4201	Magnetic Filter Funnel	005	l ea
094-219	Filter Discs, Glass	004	1 pk
	Microfiber, Reeve Angel 934-AH		(100)
D2568	Forceps, Filter	009	l ea
349-597	Pump, Vacuum, Hand Operated	004	l ea
232-413	Tongs, Crucible	004	l ea
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146706	Flask, 500 mL, Polypropylene	007	2 ea
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